

REMARKS

By the present amendment, claims 16-18 have been newly added. Claims 1-18 remain pending in the present application. Claims 1, 5 and 10 are independent claims. Applicants request reconsideration and allowance in view of the foregoing amendments and the following remarks.

35 U.S.C. § 103(a) Rejection based on admitted prior art and Moustaka

1. Claims 1, 3, 5, 6, 8, 10, 11, 13 and 15 are rejected under 35 U.S.C. § 103(a) as allegedly being unpatentable over admitted prior art in view of Moustaka (U.S. Patent No. 5,847,397). Applicants respectfully traverse this rejection.

Independent claims 1, 5 and 10 each recite a method associated with a compound semiconductor layer including nitrogen. The methods each remove part of the compound semiconductor layer by dry etching, and perform a nitrogen plasma treatment step to recover from damage due to nitrogen vacancies arising in a surface of the compound semiconductor layer as a result of the dry etching.

The present invention relates to a method of recovering from damage due to nitrogen vacancies arising in the surface of a compound semiconductor layer as a result of dry etching. In one embodiment, for example, the dry etching process is an inductively coupled plasma reactive ion etching (ICP-RIE) process carried out at room temperature (see page 11, lines 4-10). The

nitrogen plasma treatment step of the present invention is carried out in one embodiment, for example, at a substrate temperature of 40°C (see page 14, lines 27-28).

The Office Action concedes that the admitted prior art does not describe a nitrogen plasma treatment step to recover damage due to nitrogen vacancies arising in a compound semiconductor layer, and states that Moustakas teaches treating the surface with a nonetching nitrogen plasma to reduce the formation of nitrogen vacancies in col. 5, lines 39-48. The Office Action then asserts that it would have been obvious to one skilled in the art to treat the compound semiconductor with N₂ plasma because it reduces the formation of nitrogen vacancies, "which is a problem recognized by one skilled in the art at the time the invention was made."

Applicants respectfully submit that one skilled in the art at the time the invention was made would not have known the problem that dry etching would cause nitrogen vacancies. Applicants discovered this problem and then discovered the method of the invention to recover from the resulting damage. The atomic force microscope (AFM) observations and electrical measurements detailed, for example, from line 33 of page 11 to line 13 of page 14 of the present specification, led Applicants to conclude that nitrogen vacancies due to dry etching were the cause of the unwanted ohmic I-V characteristic of the gate electrode, and to seek an appropriate solution. By describing the problem that dry etching would cause nitrogen vacancies, Applicants were not implying that others skilled in the art had already recognized the problem.

An Applicant and others presented the attached paper at the 24th Electronic Materials Symposium in 2005 that reports AFM observations similar to those discussed in the present specification. These findings were presented as new information because the structure and composition of a GaN surface after treatment during the fabrication of a GaN device is still not well understood.

The Hashizume reference discussed on page 3 of the specification suggests that a nitrogen-vacancy-related state may be introduced on an H₂-plasma-treated GaN surface and that no such effect appears on N₂-plasma-treated GaN surfaces, but nothing is said about dry etching. Rather, the discussion is of epitaxial growth, followed by surface treatment at 280°C, film deposition, and wet etching with a buffered hydrofluoride solution.

Similarly, Moustakas teaches in col. 5, lines 39-48 the use of a nitrogen plasma to prevent the formation of nitrogen vacancies during a heat treatment process in which the substrate temperature is slowly raised from 270°C to 600°C, not to recover from nitrogen-vacancy damage already caused by dry etching.

As mentioned in the Amendment filed September 20, 2005, at the time the invention was made, GaN compound semiconductor materials were thought to be chemically stable and mechanically strong because of the strength of the Ga-N bond, the high crystal melting point, and the high growth temperature. It was not easy to foresee that a dry etching step carried out at a much lower temperature would produce nitrogen vacancies. The discussion of nitrogen vacancies by Moustakas in col. 4, lines 29-42 and col. 15, lines 14-20, for example, attributes

nitrogen vacancies to high-temperature crystal growth processes and teaches that growth processes at lower temperatures should reduce the number of nitrogen vacancies.

While Moustakas teaches a method of avoiding the formation of nitrogen vacancies, the claimed invention is concerned with *recovering from nitrogen vacancies that have already been formed as a result of a dry etching process*. Claims 1, 5 and 10 make this distinction clear. Applicants respectfully submit that Moustakas nowhere teaches or reasonably suggests that nitrogen vacancies might form as a result of dry etching or that nitrogen plasma treatment might be useful for recovering from such nitrogen vacancies.

Claims 1, 5 and 10 are allowable over admitted prior art and Moustakas.

Claims 3, 6, 8, 11, 13 and 15 variously depend from claims 1, 5 and 10, and are allowable as being dependent from an allowable claim.

Applicants respectfully request reconsideration and withdrawal of the rejection of Claims 1, 3, 5, 6, 8, 10, 11, 13 and 15 under 35 U.S.C. § 103(a) as being unpatentable over admitted prior art and Moustakas.

35 U.S.C. § 103(a) Rejection based on admitted prior art, Moustakas and Lee

2. Claims 2, 7 and 12 are rejected under 35 U.S.C. § 103(a) as allegedly being unpatentable over admitted prior art in view of Moustakas and Lee (U.S. Patent No. 6,762,083). Applicants respectfully traverse this rejection.

Lee fails to supplement the deficiencies of admitted prior art and Moustakas because Lee fails to teach or reasonably suggest that nitrogen vacancies might form as a result of dry etching or that nitrogen plasma treatment might be useful for recovering from such nitrogen vacancies.

Claims 2, 7 and 12 variously depend from Claims 1, 5 and 10 and are allowable as being dependent from an allowable claim.

Further, Lee describes a method for manufacturing an AlGa_N/Ga_N HFET device which is capable of easily forming a fine gate electrode. Lee fails to supplement the deficiencies of Moustakas because Lee fails to teach or reasonably suggest that nitrogen vacancies might form as a result of dry etching or that nitrogen plasma treatment might be useful for recovering from such nitrogen vacancies.

Applicants respectfully request reconsideration and withdrawal of the rejection of Claims 2, 7 and 12 under 35 U.S.C. § 103(a) as being unpatentable over admitted prior art in view of Moustakas and Lee.

35 U.S.C. § 103(a) Rejection based on admitted prior art, Moustaka and Gilbert

3. Claims 4, 9 and 14 are rejected under 35 U.S.C. § 103(a) as allegedly being unpatentable over admitted prior art in view of Moustakas and Gilbert (U.S. Patent Application Publication No. US 2002/0072223 A1). Applicants respectfully traverse this rejection.

Gilbert fails to supplement the deficiencies of admitted prior art and Moustakas because Lee fails to teach or reasonably suggest that nitrogen vacancies might form as a result of dry

etching or that nitrogen plasma treatment might be useful for recovering from such nitrogen vacancies.

Claims 4, 9 and 14 variously depend from Claims 1, 5 and 10 and are allowable as being dependent from an allowable claim.

Further, Gilbert describes a method of fabricating a ferroelectric memory device. Gilbert fails to supplement the deficiencies of Moutakas and Applicants' admitted prior art because Lee fails to teach or reasonably suggest that nitrogen vacancies might form as a result of dry etching or that nitrogen plasma treatment might be useful for recovering from such nitrogen vacancies.

Applicants respectfully request reconsideration and withdrawal of the rejection of claims 4, 9 and 14 under 35 U.S.C. § 103(a) as being unpatentable over admitted prior art in view of Moustakas and Gilbert.

New Claims

3. Newly added dependent claims 16, 17 and 18 depend, respectively, from claims 1, 5 and 10, and are allowable as being dependent from an allowable claim.

Further, these claims recite that the nitrogen plasma step is carried out at a temperature of less than 100 C, which admitted prior art, Moustakas, Lee, and/or Gilbert nowhere teach or reasonably suggest.

Conclusion

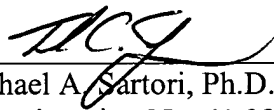
4. All of the stated grounds of rejection have been properly traversed. Applicant therefore respectfully requests that the Examiner reconsider all presently outstanding rejections

and that they be withdrawn. Applicant believes that a full and complete reply has been made to the outstanding Office Action and, as such, the present application is in condition for allowance. If the Examiner believes, for any reason, that personal communication will expedite prosecution of this application, the Examiner is hereby invited to telephone the undersigned at the number provided.

Prompt and favorable consideration of this Amendment is respectfully requested.

February 24, 2006

Respectfully submitted,

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**The 24th Electronic Materials Symposium
EMS-24**

Hotel Mielparque Matsuyama, Dogo-Himeduka, July 4-6, 2005

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K4	14:26 (3min+poster)	
Fabrication of GaN/Alumina/GaN structure to reduce dislocations in GaN		
M. Hiroki, K. Kumakura, T. Makimoto, N. Kobayashi and T. Kobayashi		
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Surface morphology of (1-101)GaN/AlGaIn/GaN heterostructure grown on (001)Si substrate by MOVPE		
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Nagoya University	237
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General model for annihilation mechanism of threading dislocations in GaN thin films grown by vapor phase epitaxy		
M. Hijikuro, S. Noguchi and N. Kuwano		
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Correlation between resistivity and luminescence intensity of GaN layers grown by MOCVD		
A. Hinoki*, Y. Hiroshima**, T. Tsuchiya**, T. Yamada**, M. Iwami**, K. Imada**, J. Kikawa**, T. Araki*, A. Suzuki*** and Y. Nanishi*		
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Photoluminescence spectra of GaN layers grown on (111)Si by CS-MBE		
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AFM and XPS characterization on annealed surface of GaN layer		
T. Makita, F. Toda and S. Seki		
Oki Electric Industry Co., Ltd.	245
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Formation of c-GaN on the surface of b-Ga ₂ O ₃ single crystalline using N ₂ plasma generated by ECR		
S. Ohira*, N. Suzuki*, J. Wada**, C. Morioka**, K. Fujiwara**, T. Yamaguchi*, T. Araki**, Y. Nanishi** and T. Shishido***		
Nippon Light Metal I*, Ritsumeikan University** and Tohoku University***	247
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Low temperature growth of cubic-GaN films on GaAs(001) substrates by electron cyclotron resonance plasma-assisted molecular-beam epitaxy		
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Osaka Institute of Technology	249
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Shutterless nitrogen flux modulation for MBE growth of nitrides using a dual-mode rf-plasma operation		
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The University of Tokyo	251
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Y. Yamamoto, T. Kondo, S. Matuoka, T. Maruyama and S. Naritsuka		
Meijo University	253

Break (14:56-15:20)

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AFM and XPS characterization on annealed surface of GaN layer

Takehiko Makita, Fumihiko Toda and Shohei Seki

Corporate Research & development Center, Oki Electric Industry Co., Ltd.

Abstract

We carried out x-ray photoelectron spectroscopy (XPS) and atomic force microscope (AFM) evaluations on surface components and structures of GaN layer after annealing in nitrogen and hydrogen atmosphere assuming ohmic sinter of GaN-HEMT fabrication process. Ga atoms desorbed from GaN surface were oxidized by 600 °C annealing process, which was lower than the growth temperature of GaN thin films. On the other hand, a part of nitrogen was desorbed from the surface and the rest possibly stayed as nitrides in the form of NH_x . The decomposition of GaN components seemed to occur nearby the GaN surfaces according to the surface roughness variation obtained from AFM images.

High-electron-mobility-transistors (HEMTs) based on GaN and related heterostructures are very promising devices in microwave field. Although there are a lot of reports on high output power of microwave using the GaN devices [1], variety of its surface structure and components after various treatments during device fabrication are still poorly understood. Especially characterization of surface after ohmic sinter process, which is performed in higher temperature, is important, because after that gate electrode is deposited on the surface, which possibly changed in quality. In this letter, we report the results of XPS and AFM evaluations of GaN surfaces before and after annealing on the assumption that ohmic sinter process was performed.

We use GaN-HEMT samples with GaN cap layer shown in Fig. 1, which grown on SiC substrate by metal-organic chemical vapor deposition (MOCVD). After cleaning the samples in an NH_4OH solution at 50 °C for the purpose of remove Ga oxide on the topmost layer of the samples [2]. The annealing temperature is fixed at 600 °C, and the samples are annealed for 0.5, 1 and 3 minutes. AFM and XPS measurement are performed before and after each annealing process. The AFM observation is carried out under the ambient air. The chemical properties of GaN surface were characterized by XPS using $\text{Al K}\alpha$ as an x-ray source.

Figs. 2 (a) and (b) show typical AFM images of the GaN surface before and after annealing for 0.5 min. in hydrogen atmosphere. The as-grown GaN exhibited smooth surface with 1 unit cell height steps and $10^8 \sim 10^9 \text{ cm}^{-2}$ of pits density. The values of the pit density kept constant after every annealing treatment. The AFM images after annealing for 0.5 ~ 1.0 min. became indistinct regardless of annealing atmosphere. This seemed to cause by microscopic structure transition of the GaN surface. AFM images came to be clear again by annealing for more than 3 min. The root-mean-square roughness (R_{MS}) values slightly increased until 1 min. of annealing time and decreased after annealing more than 1 min. of annealing time. Figs. 3(a) and (b) show the XPS intensity ratios of Ga 3d, N 1s, C 1s, O 1s normalized by total peak intensities at 0° of electron escape angle as a function of annealing time in hydrogen and nitrogen atmosphere. The XPS intensity ratios of C 1s and O 1s keep constant, on the other hand there is a tendency for Ga 3d to decrease and for N 1s to increase with

$\text{n}^+\text{-GaN (20 nm)}$
$\text{i-Al}_{0.3}\text{Ga}_{0.7}\text{N (22 nm)}$
$\text{i-GaN (2 }\mu\text{m)}$
HT-AlN buffer
n-SiC substrate

Fig. 1 Cross sectional structure of GaN HEMT sample.

increasing of annealing time. This indicates that nitrogen desorption nearby the surface of the GaN layer GaN occurs at 600°C of comparatively lower temperature to about 1100°C of growth temperature for GaN material system. In order to obtain information on the chemical component of GaN layer, we deconvoluted the XPS spectra of Ga 3d and N 1s into some components as a function of electron escape angle. XPS spectra of Ga 3d were divided into metallic Ga, GaN, Ga oxide. XPS intensity originated from Ga oxide increased with increasing of annealing time. Especially, quantity of Ga oxide after annealing in nitrogen is much larger than that of in hydrogen.

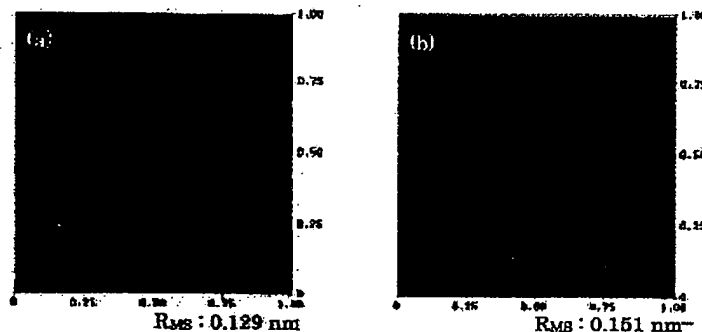


Fig. 2 AFM images of the GaN layers (a) before and (b) after annealing for 0.5 min. in hydrogen, respectively. Scan size is $1 \mu\text{m} \times 1 \mu\text{m}$.

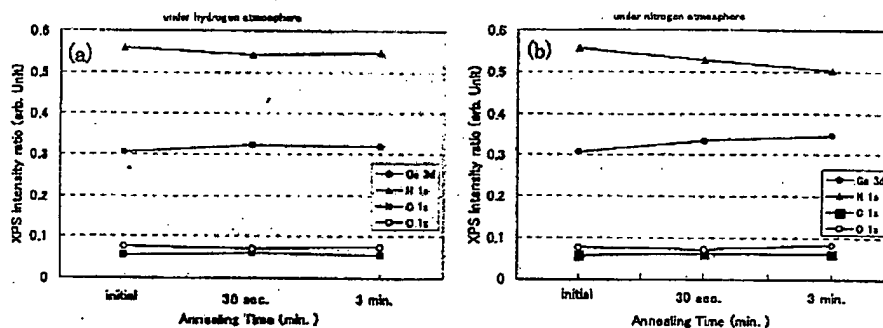


Fig. 3 XPS intensity ratios of Ga 3d, N 1s, C 1s, O 1s normalized by total peak intensities at 0° of electron escape angle as a function of annealing time in (a) hydrogen and (b) nitrogen atmosphere.

According to these results, cause of indistinct AFM images is due to GaN decomposition and consecutively nitrogen desorption and Ga oxidation, which generate from the surface to 1nm in depth of GaN layer, taking R_{MS} variation and escape depth of photo electron into consideration. We believe that these results seem to be useful for optimization of GaN-HEMT fabrication.

Reference

- [1] Y. -F. Wu, A. Saxler, M. Moore, R. P. Smith, S. Sheppard, P. M. Chavarkar, T. Wisleder, U. K. Mishra, P. Parikh, *IEEE Electron Device Lett.* 25, 117 (2004)
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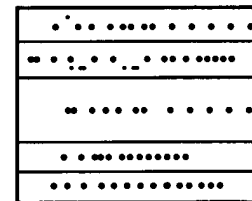


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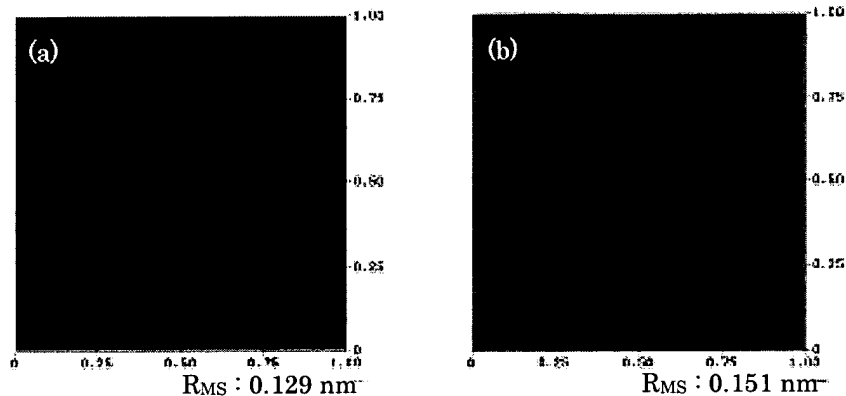


Fig. 2 AFM images of the GaN layers (a) before and (b) after annealing for 0.5 min. in hydrogen, respectively. Scan size is 1 μ m \times 1 μ m.

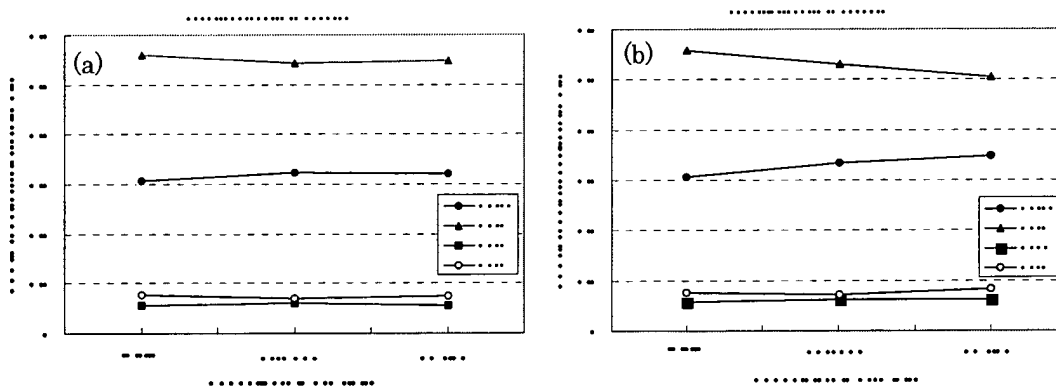


Fig. 3 XPS intensity ratios of Ga 3d, N 1s, C 1s, O 1s normalized by total peak intensities at 0° of electron escape angle as a function of annealing time in (a) hydrogen and (b) nitrogen atmosphere.

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- [2] T. Hashizume, S. Ootomo, R. Nakasaki, S. Oyama, M. Kihara, *Appl. Phys. Lett.* **76**, 2880 (2000)

24th Electronic Materials Symposium

EMS-24

EXTENDED ABSTRACTS OF THE 24th ELECTRONIC MATERIALS SYMPOSIUM



July 4th (Mon.) - 6th (Wed.), 2005

Mielparque Matsuyama, Dogo-Himmeduka

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